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IS 3598 (1983): Sodium perborate, tetrahydrate [CHD 1: Inorganic Chemicals]



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IS : 3598 - 1983

Indian Standard
SPECIFICATION FOR
SODIUM PERBORATE, TETRAHYDRATE
(*First Revision*)

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR SODIUM PERBORATE, TETRAHYDRATE (First Revision)

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Indian Standard
SPECIFICATION FOR
SODIUM PERBORATE, TETRAHYDRATE
(*First Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 28 February 1983, after the draft finalized by the Inorganic Chemicals (Misc) Sectional Committee had been approved by the Chemical Division Council.

0.2 Sodium perborate is mainly used in textile mills as a bleaching agent for a variety of fabrics, namely, wool, cotton, linen, rayon, etc, and as oxidizing and fixing agent for dyes. It is also used in dentifrices, denture cleaners and pharmaceuticals due to its cleansing, deodorizing and antiseptic properties and is incorporated in soaps and detergent powders.

0.3 The original Standard was first published in 1966. The present revision has been taken up in the light of experience gained in the manufacture of sodium perborate in the country and the end uses. Changes have been made by including the boric oxide content and specifying its limits considering the fact that various state water pollution boards have prescribed tolerance limits in the effluents. The method of packing has been changed in tune with the current practice. Keeping qualities on storage has also been revised. Colorimetric test for impurities, such as iron and copper have been suitably changed on lines with international specifications and practices.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for sodium perborate, tetrahydrate ($\text{NaBO}_3 \cdot 4\text{H}_2\text{O}$).

2. REQUIREMENTS

2.1 The material shall be white, crystalline, free flowing powder, without any foreign matter and coloured impurities.

2.2 The material when tested according to the method prescribed in Appendix A shall comply with the requirements given in Table 1. Reference to the relevant clauses of Appendix A is given in column 4 of the table.

TABLE 1 REQUIREMENTS FOR SODIUM PERBORATE, TETRAHYDRATE

Sl. No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL No. IN APPENDIX A
(1)	(2)	(3)	(4)
i)	Available oxygen, percent by mass, <i>Min</i>	10	A-2
ii)	Total alkali (as Na_2O), percent by mass	20-21	A-3
iii)	Total borate (as B_2O_3), percent by mass	22-23	A-3
iv)	Iron (as Fe), parts per million, <i>Max</i>	20	A-4
v)	Copper (as Cu), parts per million, <i>Max</i>	2	A-5

3. KEEPING QUALITY

3.1 The material when stored in a clean dry place away from heat in polyethylene lined HDPE sacks, shall meet the requirements of Table 1 for a minimum period of 3 months from the date of packing.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in polyethylene lined/clear high density polyethylene woven sacks or polypropylene or in any other mode of packing as agreed to between the purchaser and the supplier.

4.2 Marking — The packages shall be suitably marked with the following information:

- Name of material;
- Manufacturer's name and recognized trade-mark, if any;

- c) Mass of the material in the package;
- d) Month and year of manufacture; and
- e) Batch number.

4.2.1 The packages may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The procedure for drawing representative samples of the material and criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(Clause 2.2)

METHODS OF TEST FOR SODIUM PERBORATE, TETRAHYDRATE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be used in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF AVAILABLE OXYGEN

A-2.0 Principle — Available oxygen is determined by titration of a solution of the material in dilute sulphuric acid against standard potassium permanganate solution.

A-2.1 Reagents

A-2.1.1 *Dilute Sulphuric Acid* — 10 percent (v/v).

A-2.1.2 *Standard Potassium Permanganate Solution* — 0.1 N.

*Specification for water for general laboratory use (second revision).

A-2.2 Procedure — Weigh accurately about 0.3 g of the sample in a tared watch glass. Transfer to a 600-ml conical flask, containing 100 ml dilute sulphuric acid (A-2.1.1), which has been previously rendered pink by a drop of potassium permanganate solution. Titrate with 0.1 N potassium permanganate solution until the end point reaches the level of pink colour as was present before.

A-2.3 Calculation

$$\text{Available oxygen, percent by mass} = \frac{0.8 \times V N}{M}$$

where

V = volume in ml of 0.1 N potassium permanganate solution used in titration,

N = normality of potassium permanganate solution, and

M = mass in g of sample taken for test.

A-3. DETERMINATION OF TOTAL ALKALI (as Na_2O) AND BORATE (as B_2O_3)

A-3.0 Principle — Determination of the sodium oxide content by treatment of an aqueous solution of the sample with excess hydrochloric acid, followed by back titration with standard sodium hydroxide solution in the presence of methyl red as indicator. Subsequent titration of the boric oxide with standard solution of sodium hydroxide in the presence of mannitol or sorbitol and phenolphthalein as indicator.

A-3.1 Reagents

A-3.1.1 Sodium Chloride — solid.

A-3.1.2 Mannitol or Alternatively Sorbitol — neutral.

NOTE — These products shall satisfy the following test requirements: 5 g dissolved in 50 ml of carbon-dioxide free water, requires for neutralization not more than 0.3 ml of 0.02 N sodium hydroxide solution, phenolphthalein used as indicator.

A-3.1.3 Dilute Hydrochloric Acid — approximately 0.5 N solution.

A-3.1.4 Sodium Hydroxide Solution — 0.5 N free from carbonate.

A-3.1.5 Methyl Red Solution — 0.1 g/l in ethanol or rectified spirit. Dissolve 0.01 g methyl red in 95 percent (v/v) ethanol or rectified spirit (IS : 323-1959*) and make up to 100 ml with same ethanol or rectified spirit as the case may be.

*Specification for rectified spirit (revised).

A-3.1.6 Phenolphthalein Indicator Solution — 10 g/l in ethanolic solution or in rectified spirit. Dissolve 1 g of phenolphthalein in 95 percent (v/v) ethanol or rectified spirit and dilute to 100 ml with the same ethanol and rectified spirit, add a drop or two of 0.02 N sodium hydroxide solution until the first appearance of pink colour.

A-3.2 Procedure

A-3.2.1 Preparation of the Test Specimen — Weigh to the nearest 0.0005 g about 5 g of the sample, previously ground to pass through 250-microns IS Sieve and transfer to a beaker.

A-3.2.2 Add to the beaker 150 ml of water, boil gently for five minutes and digest the contents on a boiling water bath, stirring occasionally. Cool, transfer the contents to a 250-ml volumetric flask quantitatively, add 5 g of sodium chloride (**A-3.1.1**) swirl to dissolve and dilute to the mark, mix and allow the sediments, if any, to settle. Carefully drain without disturbing the sediments through a medium grade filter paper into a beaker.

A-3.2.3 Transfer by means of a pipette 50 ml of the filtrate (**A-3.2.2**), dilute with water to 100 ml, add 0.2 ml of methyl red indicator. By means of a pipette add 25 ml of hydrochloric acid. Simmer gently for 5 minutes covering the beaker with a cock glass. Allow to cool and titrate the contents against sodium hydroxide solution (**A-3.1.4**) to methyl red end point. Retain this solution for the determination of boric oxide (**A-3.4.2**).

At the same time determine the equivalence of the solutions hydrochloric acid (**A-3.1.3**) and sodium hydroxide (**A-3.1.4**) as follows:

From a pipette add 25 ml of the hydrochloric acid to 50 ml water. Add 0.2 ml methyl red (**A-3.1.5**) and titrate with sodium hydroxide solution (**A-3.1.4**).

A-3.3 To the test solution from the determination of sodium oxide content (**A-3.2.3**) add approximately 5-10 g mannitol or sorbitol (**A-3.1.2**) and 0.4 ml of phenolphthalein (**A-3.1.6**). Titrate the solution with the standard solution of sodium hydroxide (**A-3.1.4**) to a distinct pink colour.

NOTE — The solution becomes red on addition of mannitol or sorbitol on titration with sodium hydroxide, the colour of the solution turns to yellow and subsequently to the phenolphthalein pink colour end point. To ensure that the correct end point of the titration is reached, prepare a standard colour matching solution as under and compare with the solution under titration.

Mix 50 ml of a 3.8 g/l solution of disodium tetraborate decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$), 100 ml of water, 1 ml of standard hydrochloride (**A-3.1.3**), 0.2 ml of methyl red (**A-3.1.5**) and 0.4 ml of phenolphthalein (**A-3.1.6**).

A-3.4 Calculation

A-3.4.1 Sodium Oxide Content

$$\begin{array}{l} \text{Sodium Oxide (as Na}_2\text{O)}, \\ \text{percent by mass} \end{array} = \frac{V_1 - V_2}{m} \times 7.75$$

where

V_1 = volume in ml of the standard sodium hydroxide solution (A-3.1.4) used in the titration of 25 ml of the hydrochloric acid solution (A-3.1.3),

V_2 = volume in ml of the standard sodium hydroxide solution (A-3.1.4) used in the titration of the excess hydrochloride (A-3.1.3), and

m = mass in g of the test specimen.

A-3.4.2 Boric Oxide Content

$$\begin{array}{l} \text{Boric oxide content (as B}_2\text{O}_3 \text{)}, \\ \text{percent by mass} \end{array} = \frac{V_3}{m} \times 8.70$$

where

V_3 = volume in ml of the standard solution of sodium hydroxide (A-3.1.4) used after addition of mannitol or sorbitol (A-3.1.2), and

m = mass in g of the material taken for the test.

A-4. TEST FOR IRON (as Fe)

A-4.0 Principle — Iron is determined colorimetrically with orthophenanthroline, after catalytically decomposing the active oxygen, by visual comparison or by spectrophotometer/filter photometer.

A-4.1 Apparatus

A-4.1.1 Nessler Cylinders — 100 ml capacity.

A-4.1.2 Spectrophotometer — with a path length of 5 cm.

A-4.1.3 Filter Photometer — with a minimum light of 4 cm.

A-4.2 Reagents

A-4.2.1 Dilute Hydrochloric Acid — approximately 12 N.

A-4.2.2 Ammonium Hydroxide — approximately 13 N.

A-4.2.3 Potassium Iodide Solution — 5 percent (m/v).

A-4.2.4 Buffer Solution (pH_4) — Aqueous solution containing 272 g/l of crystalline sodium acetate, trihydrate is mixed with 240 ml of glacial acetic acid.

A-4.2.5 Hydroxylamine Hydrochloride Solution — 10 percent (m/v).

A-4.2.6 Orthophenanthroline Hydrochloride — 0.25 percent (m/v).

A-4.2.7 Standard Iron Solution — 20 mg/l. Dissolve 1.404 g of ferrous ammonium sulphate [$FeSO_4 (NH_4)_2 SO_4 \cdot 6 H_2O$] in about 100 ml of water containing 10-ml of dilute sulphuric acid. Dilute to 1 litre in a volumetric flask. Take 10 ml of this stock solution in a 100-ml volumetric flask and make up to volume. One ml of this diluted solution contains 0.02 mg of iron (as Fe). The dilute solution shall be freshly prepared.

A-4.3 Procedure

A-4.3.1 Using Nessler Cylinders for Colour Comparison — Weigh accurately about 50 g of the sample and transfer to a 500-ml beaker. Moisten the surface with water and by means of a pipette, introduce 100 ml of potassium iodide solution, mix well. Cover with a watch glass and decompose gently over a boiling water bath. If decomposition is excessive, cool and redecompose gently. Heat to boil for a few minutes after decomposition ceases, cool the contents to 60°C and neutralize with 12 N hydrochloric acid to a pH of 4 using a pH paper. Add 2 ml hydrochloric acid in excess to keep the solution distinctly acidic. If the temperature is kept around 50°C no precipitate shall be anticipated. Cool the solution, dilute with a little quantity of water and filter receiving the filtrate in a beaker. If any residue of boric acid is formed, wash the residue with water. Transfer the filtrate to a 500-ml volumetric flask and make up to the mark and mix well. Pipette out 10 ml of this solution into a 100 ml Nessler cylinder and develop the colour due to iron adding the reagents in the following sequence stirring each time:

1 ml of 12 N hydrochloric acid to 2 ml hydroxylamine hydrochloride and 5 ml orthophenanthroline hydrochloride to 10 ml of buffer solution. Dilute to the volume and mix well.

Carry out a control test in another Nessler cylinder with one ml of standard iron solution and the same quantities of other reagents in the same sequence and the same total volume of reaction mixture. Compare the colour of iron produced in the two cylinders.

A-4.3.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced in the test with the material is not greater than that produced in the control test.

A-4.3.3 Alternative Colorimetric Estimation Using a Spectrophotometer or Filter Photometer — If a photometer is used, measure the absorbance of the coloured solution under **A-4.1.2** against a blank of water carried through the same procedure as with the sample. Apply the blank correction and determine the iron content by reference to calibration curve, previously drawn.

A-4.3.3.1 Preparation of the calibration curve — In a series of 5 measuring flasks of 50 ml capacity, introduce respectively with the help of a pipette 0, 2, 4, 6, 8 ml of the standard iron solution (1 ml = 0.02 mg Fe). Treat the contents of each flask as follows:

Dilute to approximately 15 ml, then introduce with a pipette in the given order, mixing thoroughly each time 2 ml hydroxylamine hydrochloride (**A-4.2.5**), orthophenanthroline hydrochloride (**A-4.2.6**) and 10 ml buffer solution. Dilute to mark and mix well. After 10 minutes measure the absorbance of each solution as compared with control solution, with the help of the photometer and draw the calibration curve, against the blank at 100 percent transmittance or zero absorbance at a wavelength of 510 nm.

A-4.3.3.2 Calculation

$$\text{Iron (as Fe), parts per million} = 20 \times \frac{a}{p v}$$

where

a = mg/l of iron read on the calibration graph,

p = mass in g of the sample used, and

v = volume in ml used for colour development (**A-4.1.2**).

A-5. TEST FOR COPPER (as Cu)

A-5.0 Principle — Copper is determined colorimetrically by visual comparison using zinc dibenzyl-dithiocarbamate. The estimation can also be carried out with the aid of a spectrophotometer or filter photometer. Active oxygen does not interfere in the determination.

A-5.1 Apparatus

A-5.1.1 Nessler Cylinders — 100 ml capacity.

A-5.1.2 Buchner Flask — 250 ml with a funnel and suction pump.

A-5.1.3 Spectrophotometer (*Alternatively, Flame Photometer*)

A-5.2 Reagents

A-5.2.1 Dilute Hydrochloric Acid — 12 N, 2 N and 0.5 N.

A-5.2.2 Zinc Dibenzyl-Dithiocarbamate Solution — 0.05 percent in carbon tetrachloride (*m/v*). Preserve this solution in a brown bottle with ground glass stopper.

A-5.2.3 Standard Copper Solution — Dissolve 0.1964 g copper sulphate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in a 1 litre volumetric flask and make to volume. Pipette 10 ml of this solution into another 1 litre volumetric flask and make up to volume and mix. One ml of this solution contains 0.0005 mg copper (as Cu). The dilute solution shall be freshly prepared.

A-5.2.4 Congo Red Indicator Paper

A-5.3 Procedure (Using Nessler Cylinders)

A-5.3.1 Weigh approximately 20 g of the sample under test into a 250-ml conical flask and add 50 ml of water. After 10 minutes agitation, neutralize while shaking using 12 N hydrochloric acid in the presence of Congo red indicator paper. Add 5 ml in excess, filter through a sintered glass crucible into a Buchner flask, wash thrice with dilute hydrochloric acid, transfer all the filtrate volumetrically into a 250-ml separating funnel. Add by means of a pipette, 10 ml zinc dibenzyl-dithiocarbamate. Shake for 1 minute and allow to settle. Collect the organic layer into a 100-ml Nessler cylinder and make up to mark with carbon tetrachloride.

At the same time, prepare control test using 10 ml of standard copper solution (0.005 mg) (**A-5.2.3**) and all other reagents as the sample and develop the colour in a 100-ml Nessler cylinder.

Match the intensity of the colour produced in the two Nessler cylinders.

A-5.3.2 The limit laid down in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced with the material is not greater than that produced in the control test.

A-5.4 Procedure (Using a Filter Photometer or Spectrophotometer) — If a photometer is used, measure the absorbance of the coloured solution of sample under **A-5.3.1** against a blank of water, carried through the same procedure as the sample.

Apply the blank correction and determine the copper content by reference to the calibration curve, previously drawn.

A-5.4.1 Preparation of the Calibration Curve — In a series of four 250-ml separating funnels introduce respectively by means of a micro-burette, 0, 1, 2, 4, 6 ml of standard copper solution (**A-5.2.3**) and add the reagents in the order given below:

Dilute to 90 ml.

Acidify with 10 ml hydrochloric acid of approximately 2 N. Pipette 10 ml zinc dibenzyl-dithiocarbamate solution. Shake for 1 minute, allow to settle and let the layers separate. Collect the organic layer through a filter paper in a funnel, direct into 2-cm cell and cover immediately. Measure the absorbance of the various concentrations against the control blank and plot the calibration curve.

A-5.4.2 Calculation

$$\text{Copper (as Cu), parts per million} = \frac{a}{p}$$

where

a = mg/l of copper from the calibration curve, and

p = mass in g of the sample taken for the test.

APPENDIX B

(Clause 5.1)

SAMPLING OF SODIUM PERBORATE, TETRAHYDRATE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the sample, the material being sampled, the sampling instrument and the container for samples from adventitious contamination.

B-1.2 Samples shall be placed in clean and air-tight containers made of glass or other suitable material.

B-1.3 Each sample container shall be sealed air-tight after filling.

B-1.4 On each sample container full details of sampling, such as number of lot or batch, date of sampling and individual or composite sample, shall be marked so that the identity and source of the sample could easily be established.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In any consignment all the containers of the same size and drawn from the same batch of manufacture shall be grouped together to constitute a lot.

B-2.2 For ascertaining the conformity of the material in the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose shall depend on the size of the lot and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

NUMBER OF CONTAINERS IN THE LOT N	NUMBER OF CONTAINERS TO BE SELECTED n
(1)	(2)
Up to 25	2
26 to 50	4
51 to 100	5
101 to 300	6
301 and above	8

B-2.3 These containers shall be selected at random from the lot. To ensure the randomness of selection a random number table (see IS : 4905-1968*) may be used. In case such a table is not available, the following procedure may be adopted:

‘Starting from any container in the lot, count them as 1, 2, 3 up to r and so on in one order where r is the integral part of N/n , N being the number of containers in the lot and n being the ‘number of containers to be selected’. Every r th container thus counted shall be withdrawn to constitute the sample size.

B-3. PREPARATION OF SAMPLES

B-3.1 From each of the containers selected, draw with an appropriate sampling instrument a representative portion of the material not less than 250 g.

B-3.2 Out of the material taken from selected containers in **B-3.1** an equal quantity of material shall be taken and mixed together to form a composite sample of about 300 g. The composite sample so formed shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

*Methods for random sampling.

B-3.3 The portion of the material from each container remaining after taking the small quantity in **B-3.2** shall be divided into three equal parts, each forming an individual sample representing that particular container. There will be three sets of individual samples. Each set will contain a sample representing each selected container. One of these sets shall be marked for the purchaser, another for the supplier and the third to be used as a referee set.

B-3.4 All the individual and composite samples shall be transferred to separate moisture-proof containers and shall be labelled with full identification particulars.

B-3.5 The referee sample consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier or their authorized representatives. They shall be kept at a place agreed to between the two parties to be used in case of a dispute.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of available oxygen, total alkali and total borate shall be done on each of the individual samples.

B-4.2 Tests for all other requirements of this specification shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — The lot shall be declared as conforming to the requirements for available oxygen, total alkali and total borate if each individual sample satisfies the three requirements. In case any one of the individual samples fails in any of the three requirements, the lot shall be declared as not conforming to the requirements of this specification.

B-5.2 For Composite Sample — For declaring the conformity of the lot to the requirements of the characteristics tested on the composite sample, the test results for each characteristic shall satisfy the relevant requirement.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s ²
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²

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